

Chapter 6

Result and Conclusions

From the results obtained and analyzed various conclusions can be drawn based on each study. First of all considering the densities of the various samples it has been observed that the density of CCTO (4.93g/cm^3) ceramic prepared by the conventional solid solution technique, in the lab, is as close to any commercially available and process patented CCTO (5.07g/cm^3). The variation of density with increase in CCTO is in accordance with expected values, because both the polymers viz. PVC and PET exhibits densities much less than CCTO, and therefore the sum effect of composites is followed in this property. It is clear that the density of the composite is increased as percentage of CCTO in the composite increased. The linear dependence exhibited is graphically represented in appropriate chapter. This indicates that there is no chemical interaction between the polymer and ceramic phase. *ASTM C1323 - 10* test method was used for the other mechanical properties. X-ray diffractograms indicate that the crystalline character of the ceramic phase within the composite gets reduced with increasing percentage of Polymer. It is evident from the X-ray diffractogram of the successive samples that at higher polymer percentages the ceramic particles gets strained which in turn affects the grains leading to a broadening of the x-ray peaks. The diffractograms were indexed as well as its percentage of CCTO was confirmed. The peaks indicate the crystallite sizes of CCTO as well as polymer content. It has been observed that higher percentage of polymer in the composite has a tendency to suppress the CCTO peaks. Beyond 40% of polymers, the peak (321) is completely suppressed by the component in the composite and at 70% even (222) peak vanishes. The broadness of the peak also indicates some strain in the matrix, because grain size of CCTO is the same in all the composite series. One can therefore consider an effective grain size by calculating the apparent size using Debye-Scherrer formula¹¹².

The Scanning Electron Micrograph of the composite samples was recorded to exactly determine the connectivity of the sample¹¹³. It is very important to know the connectivity because different connectivity leads to different end results for the same phase ratios. We have assumed the material to possess 0-3 connectivity and the SEMs shall give a direct insight into the material. The SEMs of PVC, PET and CCTO ceramic in its pristine forms were first recorded, and then the subsequent composites were viewed in the light of the pristine phases and the magnification and the wavelength of the electron beam was kept uniform. The representative SEM photographs shows expected behaviour of the composites, with respect to its connectivity and morphology. It is indeed a 0-3 connectivity and the morphology is quite even. It can be observed from the micrographs that high percentages of CCTO ceramic leads to clustering of the particles leading to an effectively larger particle size and the samples shows associated effects. The effects are corroborated using X-ray studies as well as dielectric spectroscopy.

The dielectric constant and loss measurements on the composites reveals insight into the material. The pure sample of CCTO exhibits high dielectric constant at low frequency within the studied temperature range. As frequency increases, permittivity drastically decreases and approaching a constant value at 1 MHz. It has been reported that CCTO ceramics consist of insulating grain boundaries and semiconducting grains. The charge carriers accumulated at the interface between semiconducting grains and insulating grain boundaries resulted in an increase in the dielectric constant. The effect is termed interlayer boundary capacitance (ILBC). The dielectric constant of CCTO ceramics measured at different temperatures are depicted in various figures. It has been seen that there is a reduction of dielectric permittivity by two orders of magnitude just by addition of 10% polymer into the ceramic. This implies that in pure CCTO ceramic an interaction between grains or the role of grain boundaries is quite significant. This argument is all the more confirmed because further addition of polymer does not bring down the permittivity as

drastically. This in a way indirectly confirms the Inter layer barrier capacitance (ILBC) model for high dielectric constant in CCTO ceramics¹¹⁴. Dielectric loss factor of the sample exhibits dc conduction losses. It shows the dielectric loss of pure CCTO drastically decreases with increasing frequency. The dielectric loss increases with temperature from 30°C to 300°C as shown in various curves. The frequency and temperature effect on the dielectric loss illustrates the interfacial polarization of the grain boundaries within the sample. These frequency and temperature dependence are due to the interaction of phonons within the lattice. The inconsistent lattice vibrations cause instability in the interfacial polarization hence an increase in dielectric loss. Similar behavior was also reported by other researchers as well¹¹⁵.

The conductivity of the samples is typical of a material whose electrical behaviour is like an insulator with some semiconducting properties. The activation energies are typical of charge carriers with mobilities resembling ions and oxygen vacancies. The activation energies calculated from the Arrhenius plots ($\log \sigma$ vs T^{-1}) are mentioned in various figures and table, where E_a the activation energies ranging from 0.065eV to 0.217eV, are characteristic of ionic conductivity or oxygen vacancies in the bulk¹¹⁶.

6.1 Scope for future research:

The work presented in the thesis generates many more ideas which can be tested and verified for the next line of researchers in this exiting field of ceramic polymer composites. First and foremost is that this can be extended to nano domains, i.e. one may use $\text{CaCu}_3\text{Ti}_4\text{O}_{12}$ (CCTO) ceramics in its nano form but polymers with lowest possible molecular weight may be utilized. Apart from PVC and PET one can also try co-polymers with one end polar and other end non-polar i.e. co-polymer of polyvinyl alcohol and polyvinyl chloride.